

PATENT SPECIFICATION

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COMPLETE SPECIFICATION

Method of Emulsifying Highly Viscous Organopolysiloxane Oils in Water

We, THE SHIN-ETSU CHEMICAL INDUSTRY Co., LTD. of 2, 1-chome, Marunouchi, Chiyoda-ku, Tokyo, Japan a body corporate organised and existing under the law of Japan, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

10 This invention is concerned with a method of making organopolysiloxane oil-in-water (O/W) type emulsions, emulsions so made and concentrates produced as intermediate in the method.

15 Processes for dispersing hydrophobic liquid substances in water so as to make O/W emulsions of them are in wide use in industry, and various methods and apparatus have been proposed for them. For instance, it is a well-known and widely practiced method to disperse in water hydrophobic liquid substances such as oils and fats, paraffin oils and synthetic oils, together with a surface active agent and protective colloid by means of a colloid mill, Gaulin's homogenizer or a supersonic emulsifier. If the substance to be emulsified is an ordinary liquid, this method gives industrially satisfactory results.

30 However, by the method hitherto employed, it is difficult to uniformly emulsify substances such as organopolysiloxane oils, which are exceedingly hydrophobic, highly viscous, and scarcely influenced by temperature, and the emulsions obtained are apt to be unstable, so that when carrying out emulsification, a specific emulsifying agent has to be added to the oils.

40 According to the present invention, there is provided a method of emulsifying an organopolysiloxane oil having a viscosity within the range from 5,000 to 1,000,000 cs, which comprises applying strong shear to

a mixture comprising the organopolysiloxane oil, an emulsifying agent and water, by means of a roll-mill mixer equipped with at least two rollers, so as to homogenize it, and then diluting it with water, the water being present in said mixture in an amount of from 10 to 20 wt. % of said oil.

More specifically, the invention is concerned with the process of (1) adding, in the first place, an emulsifying agent and 10 to 20% by weight of water to the organopolysiloxane oil, which is hydrophobic and which can be made into an emulsion only with difficulty, (2) mixing it in a mixer or a masher, or when the quantity is small, by hand, (3) kneading the mixture with a roll-mill mixer having two, three or more rollers, the distance between whose rollers is made very narrow (e.g. under 0.1 mm) until the mixture is substantially changed and presents the appearance of an almost transparent paste, and then putting it in water.

In putting the method of the invention into practice, 10 to 20% by weight of water must be added together with an emulsifying agent to the oil, because, if the amount of water is too small, the paste produced by kneading the mixture with the rollers will not be easily dispersed in water, and when the amount is too large, it will not be easy to mix the water with the oil and the emulsifying agent. Moreover, the mixture, thus obtained, is of low concentration, thereby losing the advantage enjoyed by the above-mentioned pasty substance.

To provide the strong shear, the rollers preferably have different peripheral speeds of 5 to 20, particularly 10 to 15 metres/sec.

The addition of water to the mixture prior to homogenisation with the rollers and the dilution of the homogenised mixture with water may be carried out by any means.

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The use of a Gaulin's homogeniser or a supersonic emulsifier mentioned above will help speed up the process.

The method of the present invention is used for emulsifying organopolysiloxane oils with viscosities in the range from 5,000 to 1,000,000 cs, and more preferably in the range from 50,000 to 500,000 cs, which can be emulsified by other methods only with difficulty.

Although a wide variety of emulsifying agents can be added to organopolysiloxane oils to carry out the emulsification thereof, a good choice of emulsifying agents has much to do with the advantageous practice of the method of the invention. Emulsifying agents are of the anion type, the cation type, and the nonion type, and include polyoxyethylene-alkyl-phenol-ethers, polyoxyethylenealiphatic-alcohol-ethers, alkali metal salts and ammonium salts of fatty acids, and long-chained alkyl sulfates and alkyl sulfonates. Usually one of these is added to the oil in an amount of 1-10 wt. % based on the oil. The choice of the kind and the quantity of the emulsifying agent is made in accordance with the oil to which it is to be added. In the following Examples, detailed explanations will be given of the method of the invention, although the scope of the invention is not limited thereto.

Example 1

Methyl silicone oil, chain-stopped with trimethylsilyl group (Viscosity: 50,000 c.s./25°C) 35 wt. parts.
(Oxy ethylene)₈ nonyl phenol ether 3 wt. parts.
Water 5 wt. parts.

These ingredients were mixed well in a vessel, and the mixture was put only once through a three-roll mill mixer with the distance between the rollers made as small as possible. There was obtained a transparent paste, which was then put in 57 wt. parts of water, and placed in a shaker for 5 hours, obtaining an O/W type emulsion. This emulsion kept its stability even when it was allowed to stand for 6 months at room temperature. As a comparison, a mixture of the same ingredients was emulsified by a Gaulin's homogeniser, and the next day it was found to have separated.

Example 2

Dimethyl polysiloxane oil, chain-stopped with silanol (Viscosity: 500,000 c.s./25°C) 35 wt. parts.
(Oxy ethylene)₈ octyl phenol ether 1.2 " "
(Oxy ethylene)₈ octyl phenol ether 1.2 " "

(Oxy ethylene)₈ cetyl alcohol ether 0.6 " " 65
Water 6 " " 70

The three emulsifiers were kept at 70°C and liquefied, and water was added to them to make a mixture. Then the silicone oil was put in it, and was mixed well by means of a masher. The mixture was put twice through a three-roll mill mixer, and the paste obtained was put in 56 wt. parts of water and was emulsified by a homogeniser. The emulsion produced contained 35% oil and proved stable for more than a week at 70°C. When the emulsion was further diluted 20 times with hard water (hardness = 2), it was also found to be stable for a week at 70°C., and no separation or creaming of oil was observed. The mixture of the same ingredients could be emulsified only with difficulty by means of a homogeniser or a colloid mill, and even when the masher was used in mixing the mixture and the homogeniser was employed in emulsifying the mixture in 56 parts of water, the emulsion prepared was unstable and separated very quickly.

WHAT WE CLAIM IS:—

1. A method of emulsifying an organopolysiloxane oil having a viscosity within the range from 5,000 to 1,000,000 cs, which comprises applying strong shear to a mixture comprising the organopolysiloxane oil, an emulsifying agent and water, by means of a roll-mill mixer equipped with at least two rollers, so as to homogenize it, and then diluting it with water, the water being present in said mixture in an amount of from 10 to 20 wt. % of said oil.

2. A method according to claim 1 in which the spacing of the rollers is kept under 0.1 mm.

3. A method according to claim 1 or 2, in which the emulsifying agent is present in an amount of from 1 to 10 wt. % of said oil.

4. A method according to claim 1 substantially as hereinbefore described in either of Examples 1 and 2.

5. An emulsion when prepared by a method according to any of the preceding claims.

6. A concentrate for producing an emulsion, when prepared by the method of any one of claims 1 to 4 omitting the final dilution step.

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